

WHAT IS CLAIMED IS:

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1. A process for preparing highly esterified polyol fatty acid polyester by interesterifying polyol containing more than about four esterifiable hydroxy groups and fatty acid ester of easily removable alcohol in a heterogeneous reaction mixture wherein said easily removable alcohol is removed and said process comprises an improvement selected from the group consisting of:
- (1) the polyol is a particulate solid that has had its particle size reduced by mechanical size reduction to a particle size of less than about 100 microns;
 - (2) the process is a continuous process in which the initial catalyst level is from about 0.001 to about 0.5 mole of catalyst per mole of polyol;
 - (3) the initial stage of the reaction contains soap emulsifier at a level of from about 0.001 to about 0.6 mole per mole of polyol;
 - (4) after the degree of esterification is greater than about 60%, and the soap is insoluble in the reaction mixture, removing the soap by filtration or centrifugation in a continuous process;
 - (5) unreacted polyol having particle sizes above about one micron is removed, before any soap that is present becomes insoluble, in a continuous process;
 - (6) the molar ratio of the total ester reactant to each said esterifiable hydroxy group of said polyol in the reaction is from about 0.9:1 to about 1.4:1;
 - (7) the temperature in the initial stage of the reaction is from about 130°C to about 140°C, and in the final stages the temperature is from about 80°C to about 135°C;
 - (8) said easily removable alcohol is a volatile alcohol, the pressure is maintained at from about 5 to about 300 mm Hg and the removal of the volatile alcohol that results from the interesterification reaction is assisted by increasing the mass transfer area of the reaction mixture;

Cont.
Sub. A.

- 35 (9) the initial stage of the reaction is carried out under conditions to maintain a level of lower partial fatty acid esters of said polyol in an emulsifying amount;
- (10) at least the final stage of the reaction is carried out under conditions approaching plug-flow conditions after the degree of esterification of said polyol is at least about 50%; and
- 40 (11) mixtures thereof.

which utilizes Feature (1)

C 2. The process of Claim 1 wherein the polyol is a particulate solid that has had its particle size reduced by mechanical size reduction and has a particle size of less than about 100 microns.

H 3. The process of Claim 2 wherein said particle size is less than about 50 microns.

4. The process of Claim 3 wherein said particle size is less than about 10 microns.

5. The process of Claim 1 ~~which is a continuous process and in which the initial catalyst level is from about 0.01 to about 0.1 mole of catalyst per mole of polyol.~~

6. The process of Claim 5 in which the catalyst level is from about 0.02 to about 0.05 mole of catalyst per mole of polyol.

~~7. The process of Claim 1 wherein the initial level of soap emulsifier in the first stage of the reaction is from about 0.001 to about 0.6 mole per mole of polyol.~~

1 8. The process of Claim 7 wherein said initial level of soap emulsifier is from about 0.2 to about 0.4 mole per mole of polyol.

9. The process of Claim 8 wherein said soap emulsifier is a potassium soap of hydrogenated fatty acid containing from about 10 to about 22 carbon atoms.

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10. The process of Claim 1 wherein the process is a continuous process and in which: the initial catalyst level is from about 0.01 to about 0.1 mole of catalyst per mole of polyol; the initial level of soap emulsifier in the first stage of the reaction is
5 from about 0.0001 to about 0.6 mole per mole of polyol; and the polyol is a solid that has had its particle size reduced by mechanical size reduction and has a particle size of less than about 50 microns.

11. ~~The process of Claim 10 wherein said catalyst level is from about 0.02 to about 0.05 mole per mole of polyol; said level of soap emulsifier is from about 0.2 to about 0.4 mole per mole of polyol; and said polyol is sucrose having a particle size of less~~
5 ~~than about 10 microns.~~

12. The process of Claim 10 wherein the molar ratio of said total ester reactant to each said esterifiable hydroxy group of said polyol is from about 0.9:1 to about 1.2:1.

13. The process of Claim ^{10, 12} wherein said molar ratio of said total ester reactant to said esterifiable hydroxy group is from about 1:1 to about 1.2:1.

14. The process of Claim ⁶² 10 wherein the initial stage of said ^{interesterifying} reaction is carried out under conditions of backmixing until the average degree of esterification of the polyol is from about 20% to about 70%, ^{thereby} to provide sufficient lower partial polyol polyester
5 to aid in solubilization of the polyol.

15. The process of Claim 14 wherein the initial stage of said ^{interesterifying} reaction is carried out under conditions of backmixing until the average degree of esterification of the polyol is from about 35% to about 60%.

16. The process of Claim 15 wherein the final stages of the reaction are carried out ^{in a continuous manner} under plug-flow conditions and after the degree of esterification of said polyol is at least about 50%.

17. The process of Claim 10 wherein any soap that is insoluble in the reaction mixture is removed by filtration after the degree of esterification reaches at least about 60%. *H*

18. The process of Claim 10 wherein any unreacted polyol and catalyst having particle sizes above about one micron are removed before the degree of esterification of the polyol reaches about 75%.

a 19. The process of Claim 1 wherein the molar ratio of said total ester reactant to each said esterifiable hydroxy group of said polyol is from about 0.9:1 to about 1.2:1. *B*

20. The process of Claim 1 wherein said molar ratio of said total ester reactant to said esterifiable hydroxy group is from about 1:1 to about 1.2:1. *B*

a 21. The process of Claim 1 wherein the temperature in the initial stage of the reaction is from about 130°C to about 140°C and in the final stages is from about 80°C to about 130°C. *B*

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22. The process of Claim 21 wherein the removable alcohol is volatile and the pressure is maintained at from about 5 to about 300 mm Hg and the removal of the volatile alcohol that results from the interesterification reaction is assisted by increasing the mass transfer area of the reaction mixture.
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H 23. The process of Claim ~~21~~ ²² wherein the temperature in said initial stage is between about 132°C. and about 135°C.

H 24. The process of Claim 23 wherein said pressure is from about 15 to about 100 mm Hg.

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25. The process of Claim 1 wherein the removable alcohol is volatile and the pressure is maintained at from about 5 to about 300 mm Hg and the removal of the volatile alcohol that results from the interesterification reaction is assisted by increasing the mass transfer area of the reaction mixture.
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26. The process of Claim 25 wherein said pressure is from about 15 to about 100 mm Hg.

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27. The process of Claim 1 wherein the initial stage of the reaction contains soap emulsifier at a level of from about 0.001 to about 0.6 mole per mole of polyol and the said initial stage is carried out under conditions of backmixing to maintain a level of lower partial fatty acid esters of said polyol in an emulsifying amount.

28. The process of Claim 27 wherein said soap emulsifier is at a level of from about 0.2 to about 0.4 mole per mole of polyol and said conditions of backmixing are continued until the degree of esterification of said polyol is from about 30% to about 60%.



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29. The process of Claim 1 wherein the temperature in the initial stage of the reaction is from about 130°C to about 140°C and in the final stages is from about 80°C to about 135°C, wherein the removable alcohol is volatile and the pressure is maintained at from about 5 to about 300 mm Hg, the removal of the volatile alcohol that results from the interesterification reaction is assisted by increasing the mass transfer area of the reaction mixture; and wherein the initial stage of the reaction contains soap emulsifier at a level of from about 0.001 to about 0.6 mole per mole of polyol and the said initial stage is carried out under conditions of backmixing to maintain a level of lower partial fatty acid esters of said polyol in an emulsifying amount.

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30. The process of Claim ⁶²~~29~~ wherein the temperature in the initial stage is from about 132°C to about 135°C and the temperature in the subsequent stages is from about 100°C to about 120°C.

31. The process of Claim 30 wherein the pressure is maintained between about 15 and about 100 mm Hg.

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C6

32. The process of Claim 31 wherein said mass transfer area is increased by increasing the surface area of the reactor and/or sparging with an inert gas.

Sub A
33. The process of Claim 29 wherein said mass transfer area is increased by increasing the surface area of the reactor and/or sparging with an inert gas.

a
5 34. The process of Claim 1 wherein the initial stage of the reaction is carried out under conditions of backmixing to maintain a level of lower partial fatty acid esters of said polyol in an emulsifying amount and at least the final stage of the reaction is carried out ^{in a continuous manner} under conditions that at least approach plug-flow conditions.

Sub A
5 35. The process of Claim 34 wherein the temperature in the initial stage of the reaction is from about 130°C to about 140°C and in the final stages is from about 80°C to about 135°C, wherein the removable alcohol is volatile and the pressure is maintained at from about 5 to about 300 mm Hg, the removal of the volatile alcohol that results from the interesterification reaction is assisted by increasing the mass transfer area of the reaction mixture; and wherein the initial stage of the reaction contains soap emulsifier at a level of from about 0.001 to about 0.6 mole
10 per mole of polyol and the said initial stage is carried out under conditions of backmixing to maintain a level of lower partial fatty acid esters of said polyol in an emulsifying amount.

36. The process of Claim 35 wherein the temperature in the initial stage is from about 132°C to about 135°C and the temperature in the subsequent stages is from about 100°C to about 120°C.

37. The process of Claim 36 wherein the pressure is maintained between about 15 and about 100 mm Hg.

Sub A
38. The process of Claim 37 wherein said mass transfer area is increased by increasing the surface area of the reactor and/or sparging with an inert gas.

Sub A
39. The process of Claim 35 wherein said mass transfer area is increased by increasing the surface area of the reactor and/or sparging with an inert gas.

C
40. The process of Claim ⁶²~~34~~ wherein ~~the process is a continuous process and in which~~, the initial catalyst level is from about 0.01 to about 0.1 mole of catalyst per mole of polyol; the initial level of soap emulsifier in the first stage of the ^{interesterifying} reaction is
5 from about 0.0001 to about 0.6 mole per mole of polyol; and the polyol is a solid that has had its particle size reduced by mechanical size reduction and has a particle size of less than about 50 microns.

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41. The process of Claim 40 wherein said catalyst level is from about 0.02 to about 0.05 mole per mole of polyol; said level of soap emulsifier is from about 0.2 to about 0.4 mole per mole of polyol; and said polyol is sucrose having a particle size of less
5 than about 10 microns.

42. The process of Claim 40 wherein the molar ratio of said total ester reactant to each said esterifiable hydroxy group of said polyol is from about 0.9:1 to about 1.²:1.

43. The process of Claim ⁶²~~22~~ wherein said molar ratio of said total ester reactant to ^{each} said esterifiable hydroxy group is from about 1:1 to about 1.2:1.

44. The process of Claim ⁶²~~34~~ wherein the final degree of esterification of said polyol reaches at least about 70%.

45. The process of Claim 44 wherein the final average degree of esterification of the polyol is at least about 95%.

46. The process of Claim 1 wherein the initial stage of said reaction is carried out under conditions of backmixing until the average degree of esterification of the polyol is from about 20% to about 70%, to provide sufficient lower partial polyol polyester
5 to aid in solubilization of the polyol.

47. The process of Claim ⁶²~~46~~ wherein the initial stage of said reaction is carried out under conditions of backmixing until the average degree of esterification of the polyol is from about 35% to about 60%.

48. The process of Claim ²⁷47 wherein the final stages of the reaction are carried out ~~in a continuous manner~~ under plug-flow conditions, and after the degree of esterification of said polyol ^{has reached} is at least about 50%.

49. ~~The process of Claim 48 wherein any soap that is insoluble in the reaction mixture is removed by filtration after the degree of esterification reaches at least about 60%.~~

50. ~~The process of Claim 49 wherein any unreacted polyol, and catalyst having particle sizes above about one micron are removed before the degree of esterification of the polyol reaches about 75%.~~

51. The process of Claim ⁴⁸50 wherein ^{the} said molar ratio of said total ester reactant to ^{each} said esterifiable hydroxy group ^{of said polyol} is from about 1:1 to about 1.2:1.

52. The process of Claim 1 wherein: the molar ratio of the total ester reactant to each said esterifiable hydroxy group of said polyol in the reaction is from about 0.9:1 to about 1.4:1; the temperature in the initial stage of the reaction is from about 130°C to about 140°C, and in the final stages is from about 80°C to about 135°C; and said easily removable alcohol is a volatile alcohol, the pressure is maintained at from about 5 to about 300 mm Hg, and the removal of the volatile alcohol is assisted by increasing the mass transfer area of the reaction mixture.

53. The process of Claim 52 wherein unreacted polyol having particle sizes above about one micron is removed before any ^{soap} soap that is present becomes insoluble and the initial catalyst level is from about 0.001 to about 0.5 ⁵ mole per mole of polyol.

54. The process of Claim 1 which is carried out in a series of at least two reaction vessels.

55. The process of Claim 54 wherein there are from three to about eight of said reaction vessels.

Sub C
56. The process of Claim 1 wherein: the temperature in the initial stage of the reaction is from about 130°C to about 140°C and in the final stages is from about 80°C to about 135°C; the removable alcohol is volatile and the pressure is maintained at
5 from about 5 to about 300 mm Hg; the removal of the volatile alcohol that results from the interesterification reaction is assisted by increasing the mass transfer area of the reaction mixture; the initial stage of the reaction contains soap emulsifier at a level of from about 0.001 to about 0.6 mole per mole of
10 polyol; said initial catalyst level is from about 0.01 to about 0.1 mole per mole of polyol; the molar ratio of said total ester reactant to each said esterifiable hydroxy group of said polyol is from about 0.9:1 to about 1.4:1; the polyol is sucrose having a particle size of less than about 100 microns; the unreacted
15 sucrose is removed after the degree of esterification is at least about 15% and before the soap becomes insoluble; the said initial stage is carried out under conditions of backmixing to maintain a level of lower partial fatty acid esters of said polyol in an emulsifying amount; and the final stages are carried out under
20 plug flow conditions after the degree of esterification is at least about 50%.

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57. The process of Claim ⁵⁶~~56~~ wherein the soap emulsifier is removed after the degree of esterification is at least about 60%.

c
58. The process of Claim 1 wherein any unreacted polyol and catalyst having particle sizes above about one micron are removed while the soap emulsifier is soluble in the reaction mixture and before the degree of esterification of the polyol reaches about
5 75%, and wherein the temperature in the initial stage of the reaction is from about 130°C to about 140°C and in the final stages is from about 80°C to about ~~135°C~~
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59. The product prepared by the process of Claim 1.

60. The product prepared by the process of Claim 52.

add
C1 x D1
61. The product prepared by the process of Claim 53.